

# Microstructural Evolution in the Intercritical Heat Affected Zone of a Boron Containing Modified 9Cr-1Mo Steel

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## Abstract

Type IV cracking observed in high Cr ferritic steels is attributed to poor creep properties of the intercritical heat affected zone (ICHAZ) of the weld joint which in turn associated with the partial transformation that take place in this zone during the weld thermal cycle and resulting refinement of the microstructure. Recent studies on steels with controlled addition of boron has shown that creep strength of ICHAZ in these steels are comparable to that of the base metal and the microstructure of this zone is significantly different from that of the steels with out boron. Hence in this paper microstructural evolution in the simulated ICHAZ of two different P91 steels, one without boron and another with boron and reduced nitrogen content has been studied. Results indicate that during heating part of the weld thermal cycle, austenite nucleate and grow into two different morphologies, globular and acicular which transform to martensite during cooling. The former is more prevalent along the prior austenite grain boundaries (PAGB) of the base metal while the latter along the lath boundaries of the tempered martensite within the grains. Results also show the transformation to austenite is delayed in the boron containing steels and austenite with both the morphologies are formed. However, growth of the globular austenite, formed along the PAGB is sluggish and hence PAGBs are discernable even after the transformation. This explains the difference in the microstructure of the of ICHAZ of steels with boron and without boron and this could also be the reason for the improved creep resistance of the weld joint of high Cr ferritic steels with controlled addition of boron.

## Introduction

Modified 9Cr-1Mo steel is used extensively in fossil power plants for its superior creep and oxidation resistance at service temperature and good thermo physical properties. This steel is used in the normalised and tempered condition for good creep strength and ductility. Mechanical properties are closely related with initial microstructure of material. The attainment of good mechanical properties lies with selection of proper heat treatment temperature. Low temperature normalizing treatment provides uniform and fine austenite grain size whereas high temperature heat treatment provides non uniform and coarse austenite grain

microstructure. The former heat treatment provides good strength and ductility whereas latter heat treatment results in low ductility; but can result in an ICHAZ in the weld joints that is more resistant to Type IV cracking. Therefore, achieve optimum properties an intermediate heat treatment temperature at which most of the precipitates dissolve and the austenite does not coarsen.

Though optimum mechanical properties can be achieved in the base metal, life of the components is dependent on weld joints strength and ductility, which is in turn dependent on the composition of the base metal and weld metal, welding parameters and heat treatment to which the joints are subjected to [1]-[2]. It has been reported that life of the weld joint is lower than that of the base metal and in service or during creep test at low stress and high temperatures they fracture at ICHAZ, which is referred as Type IV cracking [3]. To circumvent this problem, 9Cr-3W-3Co-V-Nb ferritic steel with controlled addition of boron and nitrogen has been developed by NIMS, for which the creep strength of the weld joint is reported to be comparable to that of the base metal [4]. Enhancement of creep strength has been attributed to the stability of the microstructure at the ICHAZ; which is caused by the presence of boron though the exact mechanism is not yet clearly understood [5].

It has been also seen austenite with two different morphologies are formed when ferritic steels are heated to intercritical temperature range. Kimmins and Gooch [6] has observed austenite nucleation both along the prior austenite boundaries (grows into globular morphology) and within the grains (acicular morphology) in normalized 1Cr-1Mo-0.75V(Ti,B) steel when heated to temperature between  $AC_1$  and  $AC_3$ . Many authors have attributed that nucleation of acicular austenite is associated with films of retained austenite [6] on lath boundaries and lath stability of prior microstructure determines the growth morphology. They also reported 4-5% retain austenite in their material.

In the present investigation microstructural evolution in the ICHAZ of two steels of nominal composition of P91 steel, one without boron and another with boron and reduced nitrogen content is compared by subjecting the steel specimens to simulated heat treatments that produce ICHAZ microstructure.

## Experimental

As received modified 9Cr-1Mo steels with and without boron has been designated as P91 and P91B are normalized at 1050, 1100 and 1150°C for 1 h. Subsequent to normalizing they have been tempered at 760°C for 3 h followed by furnace cooling. ICHAZ microstructure was simulated in these normalised and tempered (N&T) steels by holding these steels at preset temperature of 875 and 900°C for different durations of 5, 10 and 30 minutes, followed by water quenching. These simulated specimens were then tempered at 760°C for 3 h (simulated post weld heat treatment) followed by furnace cooling. Specimens were cut into 10×10×3 mm and polished metallographically up to 0.25 μm in diamond slurry. Hardness was measured at 10 kg load using Vicker's hardness tester. Polished specimens were etched using Vilella's reagent for microstructural studies.

## Results and Discussion

### Microstructure of Base Metal

It is known that the PAGS of P91 and P91B increases with increasing normalizing temperature [7]-[8]. However, results from these two steels show that this increase is higher at 1150°C for P91B than P91. PAGS of these two steels after different normalizing temperatures is given in Table 1. The grain size was estimated using linear intercept method using image analysis software. For the estimation, average of 50 grains was taken at single magnification. From the table it is clear that grain size increases with increasing normalizing temperature. The large grains in P91B can be attributed to lower nitrogen content. The chemical compositions of these steels are given in Table 2.

TABLE 1: PRIOR AUSTENITE GRAIN SIZE OF P91 AND P91B STEEL WAS ESTIMATED AFTER NORMALIZING AND TEMPERING (AT 760°C FOR 3 HOURS) TREATMENT.

Heat Treatment Condition	P91	P91B
1050°C/1h	20±10μm	30±10μm
1100°C/1h	100±20μm	150±30μm
1150°C/1h	150±50μm	250±50μm

TABLE 2: CHEMICAL COMPOSITION OF P91 AND P91B

Elements	C	Cr	Mo	Si	Mn	V	Nb	S
P91 B	0.1	8.5	1.04	0.40	0.3	0.23	0.09	0.002
P91	0.1	9.5	1.0	0.48	0.39	0.25	0.1	0.009
Elements	P	Ni	Al	Ti	N	B	Cu	Fe
P91 B	0.005	0.02	0.03	0.003	0.0021	0.0100	0.006	Bal.
P91	0.021	0.14	0.024	-	0.0650	-	-	Bal.

Except for addition of boron and low nitrogen content in P91B steel, the compositions are similar. Low nitrogen content decreases volume fraction of MX precipitates which being stable even at about 1150°C effectively retards the grain growth [9]. Hence, with low nitrogen content in P91B grain growth is more in this than in P91. The microstructure of P91 and P91B for 1150°C/1 h normalizing and 760°C/3 h tempering are shown in Figure 1.

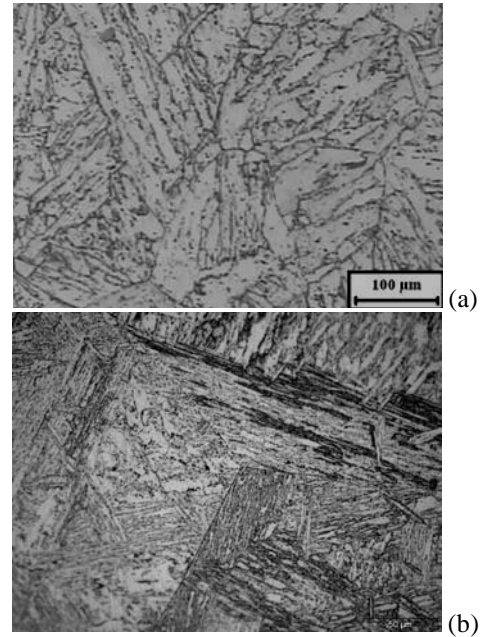


Figure 1: Microstructure of (a) P91 and (b) P91B normalised at 1150°C for 1 h and tempered at 760°C for 3 h

These microstructures also show lath structure is finer in P91B than in P91. Scanning electron micrographs consists of lath martensite with precipitates decorating lath and PAGB (Figure 2) in both steels [7]-[8]. From these it is clearly evident that laths and precipitates are finer in the former than the latter. These precipitates were found to be predominantly  $M_{23}C_6$  in P91B and  $M_{23}C_6$  and MX in P91. Microstructure of P91 and P91B normalised at 1050 and 1100°C for 1 h are also similar.

### Microstructural Evolution in the ICHAZ

As the prior austenite grain size significantly influences the ICHAZ of the ferritic steels and major difference in the microstructural features of steels with boron and without boron has been reported, these features are carefully examined in the simulated HAZs of both the steels. Effect of initial grain size, peak simulation temperature and hold time on the simulated ICHAZ microstructure was examined.

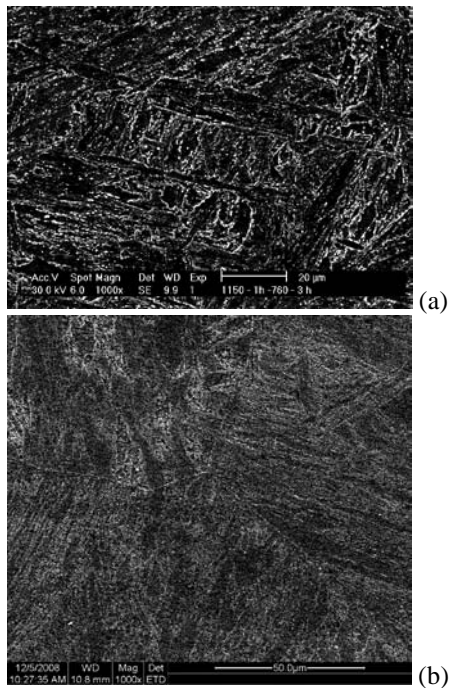


Figure 2: Normalised (1150°C/1 h) and tempered (760°C/3 h) microstructure of (a) P91 and (b) P91B

As seen from Table 1, P91 and P91B heat treated at 1050 and 1100°C for 1 h has smaller prior austenite grain size than those heat treated at 1150°C. Microstructure of ICHAZs simulated for different durations of these specimens indicated that austenite with two different morphologies, as reported by Kimmins and Gooch [6] forms during heating part of the ICHAZ simulation. In P91, with small PAG size, the austenite nucleate predominantly along the PAGB and morphology of the austenite is globular. However, with large PAG size (normalized at 1150°C) austenite transformation begins within the grains (acicular austenite according to Kimmins and Gooch [6]) and during subsequent transformation, this forms lath martensite. Figure 3(a) shows microstructure of the P91 (normalized at 1150°C/1 h) ICHAZ simulated at 875°C for 5 minutes. In contrast to this, microstructure of simulated ICHAZs of P91B with small prior austenite grain size also show lath martensite. Further, such a transformation during ICHAZ simulation retained original austenite grain boundaries. Figure 3(b) shows the microstructure of the ICHAZ of P91B normalized at 1100°C/1 h and simulated at 900°C/5 minutes.

Effect of hold time at the peak temperatures of simulation (875 and 900°C) for both steels normalised at 1150°C for 1 h and tempered at 760°C for 3 h were also studied separately. The micrographs for 875°C simulation for different duration of hold time for both the steels are shown in Figure 4. They show grain size in simulated ICHAZ has

refined significantly in P91 irrespective of hold time. In comparison with prior austenite grain size of 150µm, the average grain size of the ICHAZ after 30 minutes of hold time at peak temperature of simulation is only  $20 \pm 5 \mu\text{m}$ . This clearly confirms that grain refinement has taken place in P91 during simulation. Micrographs indicate austenite formed in P91 during simulation is of globular morphology. As the austenite of this morphology is nucleated along the grain boundaries, PAGBs present prior to simulation are no longer discernable in the micrographs. In contrast, microstructural variation of simulated ICHAZ of P91B with duration of simulation is vastly different. From the micrographs (Figure 5) it is apparent that no transformation has taken place for a hold time of 5 minutes. However, there is evidence of transformation after 10 minutes; the lath boundaries are fine and the PAGBs present before simulation decorated with new transformation products. The new martensite laths and the transformation products found along the PAGB are formed during simulation heat treatment. As already explained, during simulation austenite with globular morphology was formed along the PAGBs and that with acicular morphology within the grains. Increasing the duration of simulation heat treatment increased the volume fraction of the transformation products as clearly seen in the microstructure of the specimen subjected to simulation for 30 minutes.

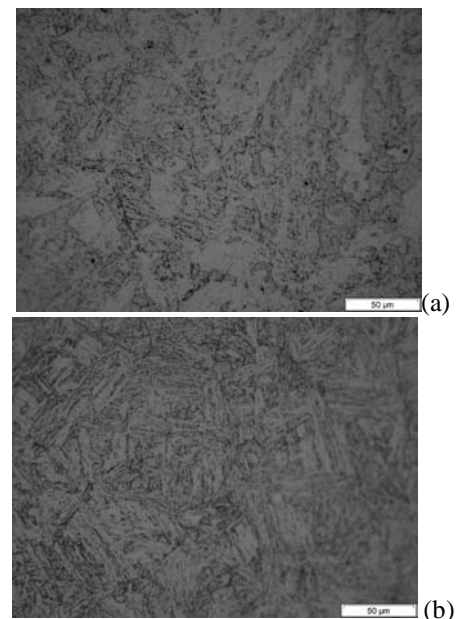


Figure 3: (a) Simulated ICHAZ (875°C/5 minutes) P91 (1150°C/1 h and 760°C/3 h), (b) Simulated ICHAZ (900°C/5 minutes) P91B (1100°C/1 h and 760°C/3 h)

Tabuchi et al [10] also has reported such retention of original prior austenite grain boundaries in P92 steel during simulation of ICHAZ microstructure. However, they did

not temper the material before giving simulation treatment. However, electron back scattered diffraction (EBSD) and TEM studies did not reveal any retained austenite in the material. With increasing holding time the transformation along the grain boundaries was found to be increasing. The construction of original grain is referred as austenite memory effect in literature [6].

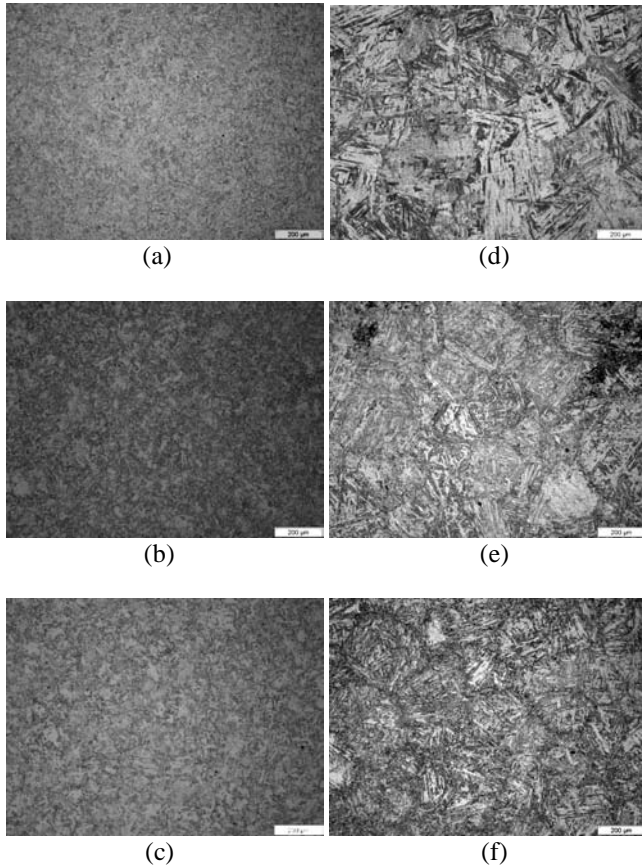


Figure 4: Effect of hold time at peak simulation temperature (875°C) for microstructure of ICHAZ of (a, b, c) P91 and (d, e, f) P91B for hold times of 5 minutes (a, d), 10 minutes (b, e), and 30 minutes (c, f)

In line with 875°C simulation, microstructures observed in both the steels after simulation at 900°C were similar. P91 did show similar grain refinement, with a PAGS slightly larger than that is observed after simulation at 875°C. In P91B, there is evidence to show that austenite has nucleated at PAGB even after 5 minutes of simulation at 900°C which was not observed after 875°C simulation. Microstructure of simulated ICHAZ of P91B (1150°C/1h) after simulation at 900°C for 5 and 30 minutes are shown in Figure 5. This suggests critical time is required for austenite to nucleate at grain boundaries (globular austenite) decreases with increase in the temperature of simulation.

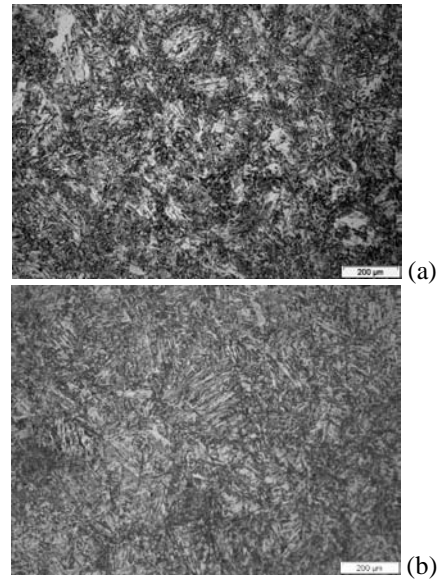


Figure 5: Effect of hold time at peak simulation temperature (900°C) for microstructure of ICHAZ of P91B (a) 10 minutes and (b) 30 minutes

From the results presented and discussed above, it is clear that presence of boron in the steel significantly alter the transformation behaviour. The transformation temperature ( $A_{c1}$ ) increases with boron addition, kinetics of transformation is slow and morphology of the austenite formed along the PAGB and within the grain seems to be different. All these significantly affect the evolution of the microstructure in the ICHAZ of the boron steel. PAGB of the base metal is discernable and lath structure is maintained even in the transformed products formed during ICHAZ simulation treatment. PAGB is also decorated with fine globular austenite formed during simulation which does not grow as in boron free steel. It is reasonable to assume that these microstructural features have significant influence on the improved creep resistance of the weld joints of boron containing high Cr steels. As no retained austenite was observed either in P91 and P91B, present study did not find any evidence for retained austenite in retaining the prior austenite grain size and boundaries even after transformation (often referred as austenite memory effect) in the boron steel.

## Conclusions

- (1) Presence of boron significantly alters the microstructural evolution in the ICHAZ of the P91 steel
- (2) Austenite with two different morphologies –globular along the grain boundaries and acicular within the grains- is formed during ferrite to austenite transformation. In boron steels growth of the globular austenite is sluggish in boron containing steel and hence the PAGB are retained even after transformation.

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